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Sung R. Choi
Ohio Aerospace Institute, Brook Park, Ohio

Narottam P. Bansal and John P. Gyekenyesi
Glenn Research Center, Cleveland, Ohio

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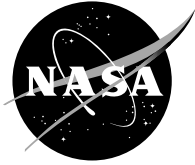
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Ohio Aerospace Institute, Brook Park, Ohio

Narottam P. Bansal and John P. Gyekenyesi
Glenn Research Center, Cleveland, Ohio

National Aeronautics and
Space Administration

Glenn Research Center

Acknowledgments

This work was supported in part by the UEET Program at NASA Glenn Research Center. The authors are grateful to R. Pawlik for the experimental work during the course of this work.

This report is a formal draft or working paper, intended to solicit comments and ideas from a technical peer group.

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Sung R. Choi
Ohio Aerospace Institute
Brook Park, Ohio 44142

Narottam P. Bansal and John P. Gyekenyesi
National Aeronautics and Space Administration
Glenn Research Center
Cleveland, Ohio 44135

ABSTRACT

Both interlaminar and in-plane shear strengths of a unidirectional Hi-Nicalon™ fiber-reinforced barium strontium aluminosilicate (SiC/BSAS) composite were determined at 1100 °C in air as a function of test rate using double notch shear test specimens. The composite exhibited a significant effect of test rate on shear strength, regardless of orientation which was either in interlaminar or in in-plane direction, resulting in an appreciable shear-strength degradation of about 50 percent as test rate decreased from 3.3×10^{-1} mm/s to 3.3×10^{-5} mm/s. The rate dependency of composite's shear strength was very similar to that of ultimate tensile strength at 1100 °C observed in a similar composite (2-D SiC/BSAS) in which tensile strength decreased by about 60 percent when test rate varied from the highest (5 MPa/s) to the lowest (0.005 MPa/s). A phenomenological, power-law slow crack growth formulation was proposed and formulated to account for the rate dependency of shear strength of the composite.

INTRODUCTION

The successful development and design of continuous fiber-reinforced ceramic matrix composites (CFCCs) are dependent on understanding of their basic properties such as deformation, fracture and delayed failure (fatigue, slow crack growth, or damage accumulation) behavior. Particularly, accurate evaluation of delayed failure behavior under specified loading/environment conditions is prerequisite to ensure accurate life prediction of CFCC structural components at elevated temperatures.

Although CFCCs have shown improved resistance to fracture and increased damage tolerant as compared to monolithic counterparts, inherent material/processing defects or cracks in the matrix-rich interlaminar regions can still cause delamination by the presence of interlaminar normal or shear stress, leading to loss of stiffness or in some cases possible structural failure. Strength behavior of CFCCs in shear has been characterized in view of their unique interfacial architectures and its importance in structural applications [1–5]. Due to inherent nature of *ceramic* matrix composites, it would be highly feasible that interlaminar defects or cracks are susceptible to slow crack growth or damage accumulation in certain environments (mostly air) particularly at elevated temperatures, resulting in strength degradation or time-dependent failure. Although slow crack growth is one of important life-limiting phenomena, little studies have been done on this subject of slow crack growth of CFCCs under *shear* at elevated temperatures.

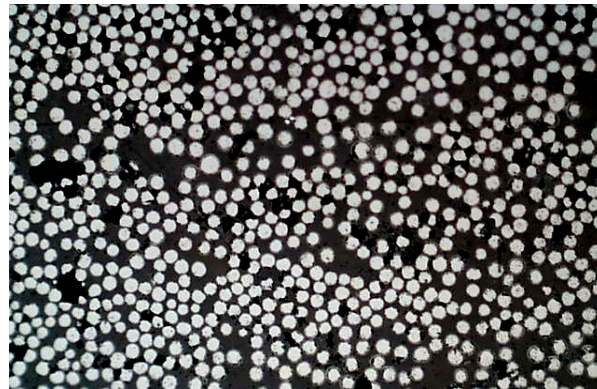
In the previous studies [6,7], ultimate *tensile* strength of several different CFCCs was determined as a function of test rates at elevated temperatures in air ranging from 1100 to 1200 °C. It has shown that the ultimate tensile strength of those CFCCs depended significantly on test rate: the greater strength at the greater test rate, and vice versa. The leading mechanism has been understood as slow crack growth or damage accumulation, evidenced additionally by the results of both stress rupture and preloading testing.

This has suggested that life prediction parameters of CFCCs could be estimated at least for a short range of lifetimes by a relationship between ultimate tensile strength and test rate, termed “dynamic fatigue” that has been used in many monolithic brittle materials including glasses, glass-ceramics, and advanced ceramics. The current work, extending the previous studies to include shear, was to determine the rate dependency of *shear* strength at 1100 °C in air using a unidirectional Hi-Nicalon™ fiber-reinforced barium strontium aluminosilicate (SiC/BSAS) composite. Both interlaminar and in-plane shear strengths of the SiC/BSAS composite were determined with double-notch shear test specimens as a function of test rate ranging from 3.3×10^{-5} to 3.3×10^{-1} mm/s. The effect of test rate on shear strength of the 1-D SiC/BSAS composite was compared with that on tensile strength of a similar 2-D SiC/BSAS composite. A phenomenological, slow crack growth model was proposed to describe the rate-dependency effect of shear strength of the composite.

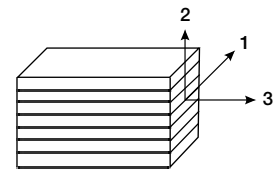
EXPERIMENTAL PROCEDURES

The processing of SiC/BSAS composite can be found elsewhere [5,8]. Hi-Nicalon fibers with an average diameter of 14 μm were used as reinforcement. The fiber surfaces were coated with 0.4 μm BN followed by 0.1 μm SiC by chemical vapor deposition. The BN interfacial layer acts as a weak, crack deflection phase, while the SiC overcoat acts as a barrier to diffusion of boron from BN into the oxide matrix and also prevents diffusion of matrix elements into the fiber [8]. The precursor to the celsian matrix of 0.75BaO-0.25SrO-Al₂O₃-2SiO₂ (BSAS) was made by solid-state reaction. The precursor powder consisted of mainly SiO₂ and BaAl₂O₄ with small amounts of Ba₂SiO₄, α-Al₂O₃, and Ba₂Sr₂Al₂O₇. This powder was made into a slurry with an organic solvent with various additives. Tows of BN/SiC-coated fibers were impregnated with the matrix precursor by passing them through the slurry. The resulting prepreg tape was dried and cut into pieces. Unidirectional fiber-reinforced composite was prepared by tape lay up (20 plies) followed by warm pressing at 150 °C to form a “green” composite. Finally, dense composites were obtained by hot pressing under vacuum in a graphite die. X-ray diffraction showed that the precursor was fully converted into the desired monoclinic celsian phase through solid-state reaction. The fully dense composite laminate was about 4.2 mm thick and had a fiber volume fraction of about 0.42. Typical examples of microstructure of the SiC/BSAS composite in planes parallel and perpendicular to the fiber direction are shown in Figure 1.

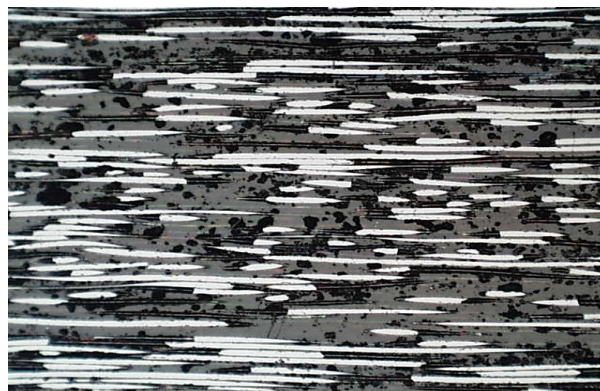
The double-notch shear (DNS) test specimens measuring 4 mm (width) × 4 mm (depth) × 20 mm (length), as shown in Figure 2, was machined from the composite laminate. The DNS test specimens were used previously for the determination of interlaminar and in-plane shear strength of the composite at both ambient and elevated temperatures [5]. The square cross-section was intentionally made to make a one-to-one comparison between the interlaminar and in-plane shear strength at a given test condition without the interference of possible size effect. Dimensions of DNS test specimens used in this work were different from those recommended by ASTM C 1425 [4] since the test specimens in the standard has a rectangular cross section and would not be appropriate to determine both interlaminar and in-plane shear strength. Two notches were 0.3 mm wide, 5 mm away from each other, and situated in equal distance from both ends. The two notches were extended exactly to the middle of each specimen so that failure occurs on the plane between the notch tips. Detailed descriptions and stress analysis of the DNS test specimens can be found elsewhere [5]. Monotonic shear testing was conducted in ambient air at 1100 °C for the SiC/BSAS DNS specimens using an electromechanical test frame (Model 8562, Instron, Canton, MA). A simpler test-fixture configuration consisting of SiC upper and lower fixtures, as shown in Figure 3, was used due to the merits of test specimen’s configuration as well as its tight machining tolerances, with which test specimen could stand alone - a free-standing feature. A total of five test rates ranging from 3.3×10^{-5} to 3.3×10^{-1} mm/s were used in displacement control in both interlaminar and in-plane shear testing. Typically, three specimens were used at each test rate for a given material direction. Each test specimen was held for about 20 min at the test temperature for a thermal equilibration prior to



(a) Plane ('1-2') normal to fiber direction



(b) Plane ('3-2') parallel to fiber direction



(c) Plane ('3-1') parallel to fiber direction

Figure 1. Microstructure of unidirectional (1-D) SiC/BSAS ceramic matrix composite: (a) normal to fiber direction, (b) and (c) parallel to fiber direction. The insert defines material's axes and planes.

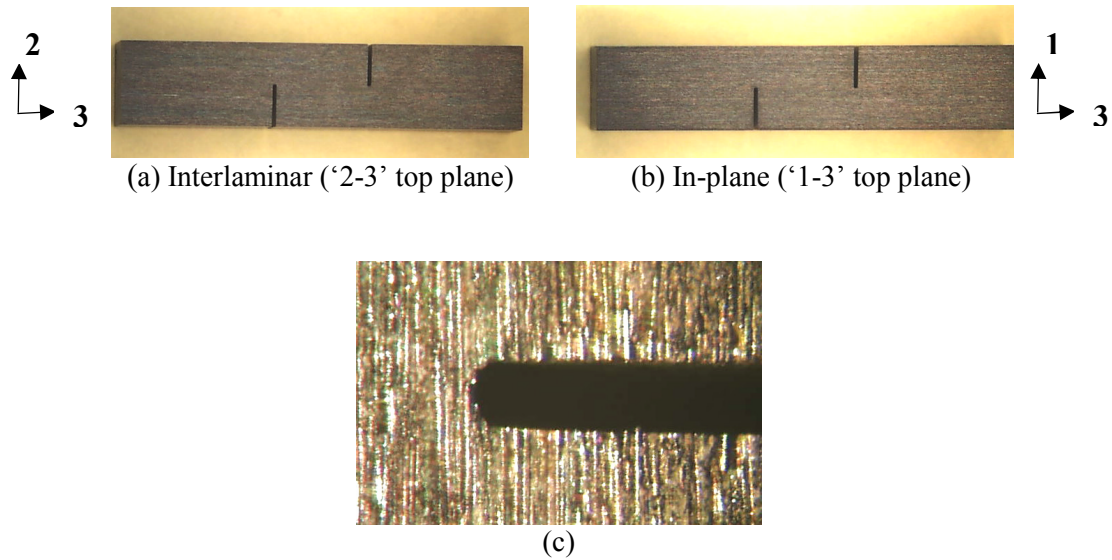


Figure 2. Double notch shear (DNS) test specimens [5] used this work showing overall views of interlaminar (a), in-plane (b) test specimens, and notch detail (c). The material's axes and planes are defined in the insert of Figure 1.

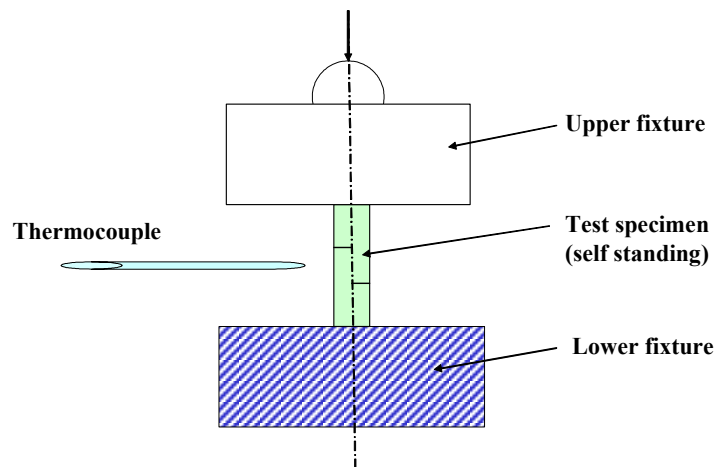


Figure 3. A schematic showing test fixture and test specimen used in this work.

testing. The shear fracture stress, average shear stress at failure, was calculated using the following relation

$$\tau_f = \frac{P_f}{WL_n} \quad (1)$$

where τ_f is the shear strength, P_f is the fracture load, W and L_n are the specimen width and the distance between the two notches, respectively. A limited fractographic analysis was performed in an attempt to help understand mechanisms associated with shear failure.

EXPERIMENTAL RESULTS

The results of monotonic shear strength testing for the SiC/BSAS composite are presented in Figure 4, where *shear strength* was plotted as a function of *applied test rate* for both interlaminar and in-plane directions using a log-log scale. Each solid line in the figure represents a best-fit regression line based on the log (*shear strength*) versus log (*applied test rate*) relation (The reason for using the log-log relation will be described in the ‘Discussion’ section.). The decrease in shear strength with decreasing test rate, termed a susceptibility to slow crack growth or damage accumulation (or delayed failure), was significant for either interlaminar or in-plane direction. The shear strength degradation was about 48 and 59 percent, respectively, for interlaminar and in-plane directions when test rate decreased from the highest (3.3×10^{-1} mm/s) to the lowest (3.3×10^{-5} mm/s). For a given test rate, in-plane shear strength was about 70 percent greater than interlaminar shear strength, an important factor when a conservative design is considered and sought. The interlaminar plane was relatively easier in cleavage in either shear or tension than the in-plane counterpart at elevated temperature, attributed to the nature of the composite’s tape lay-up (laminated) architecture and of the weakened ‘glassy’ (aluminosilicate) matrix due to high temperature. Note that except for the magnitude of shear strength, the trend in strength degradation with respect to test rate was basically the same for both interlaminar and in-plane directions. The shear-strength degradation was analogous to the trend of tensile strength with respect to test rate that has been observed in various CFCCs including SiC/MAS, SiC/CAS, SiC/BSAS, C/SiC, and SiC/SiC composites [6,7]. These CFCCs have exhibited significant degradation of tensile strength with decreasing test rates, with their degree of degradation being dependent on material and test temperature.

Figure 5 shows typical load-versus-time curves determined at two different test rates for both interlaminar and in-plane test specimens. At a fast test rate of 3.3×10^{-2} mm/s, an initial settling stage was followed by a linear region until the maximum load reached, beyond which a typical composite failure mode was followed. At a much slower test rate of 3.3×10^{-4} mm/s, the applied load increased *linearly* up to the peak load and dwelled at the peak a little while followed by a sudden drop. The dwelling of the peak load was clearly an indication of a phenomenon associated with slow crack growth. At faster test rates, a test specimen was subjected to shorter test time so that a crack had no enough time to grow, resulting in insignificant slow crack growth or little strength degradation. By contrast, at a slower test rate, a crack was subjected to longer time so that a crack had enough time for slow crack growth, thereby yielding significant strength degradation. Creep was insignificant even at lower test rates, as can be seen from the *linear* behavior of load versus time curves. The insignificant creep in shear for the 1-D SiC/BSAS composite was in contrast to the somewhat significant creep in tension at lower test rates by a similar 2-D SiC/BSAS composite tested at 1100 °C in air [7].

Figure 6 shows fracture surfaces of both interlaminar and in-plane specimens tested at high (3.3×10^{-1} mm/s) and low (3.3×10^{-5} mm/s) test rates. The composite, regardless of material direction, did not exhibit any overall, noticeable signs of slow crack growth from their fracture surfaces. Also, the fracture surfaces showed little difference in the mode and feature of fracture between high and low test rates. However, fracture surfaces were generally smoother in interlaminar than in in-plane specimens.

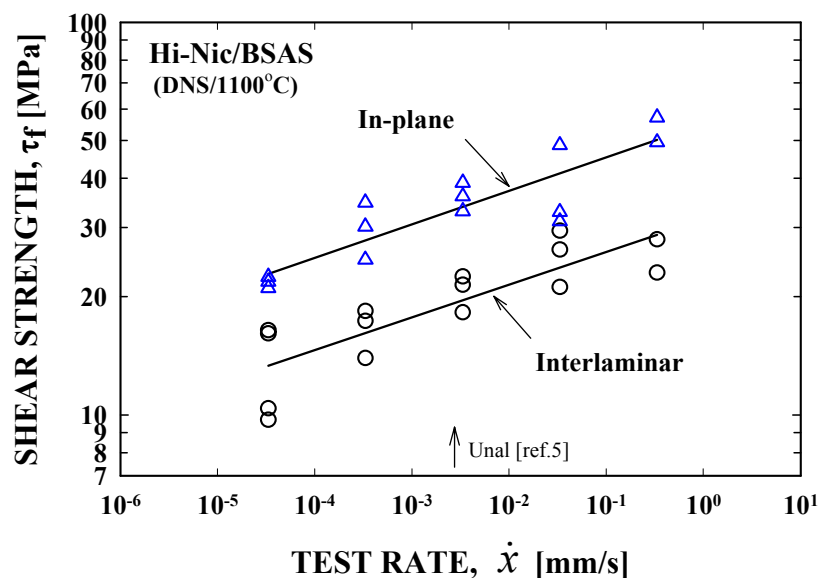


Figure 4. Shear strength as a function of test rate in both interlaminar and in-plane directions for SiC/BSAS composite tested at 1100 °C in air. The arrow in the ‘test-rate’ axis indicates the test rate used by Ünal and Bansal [5].

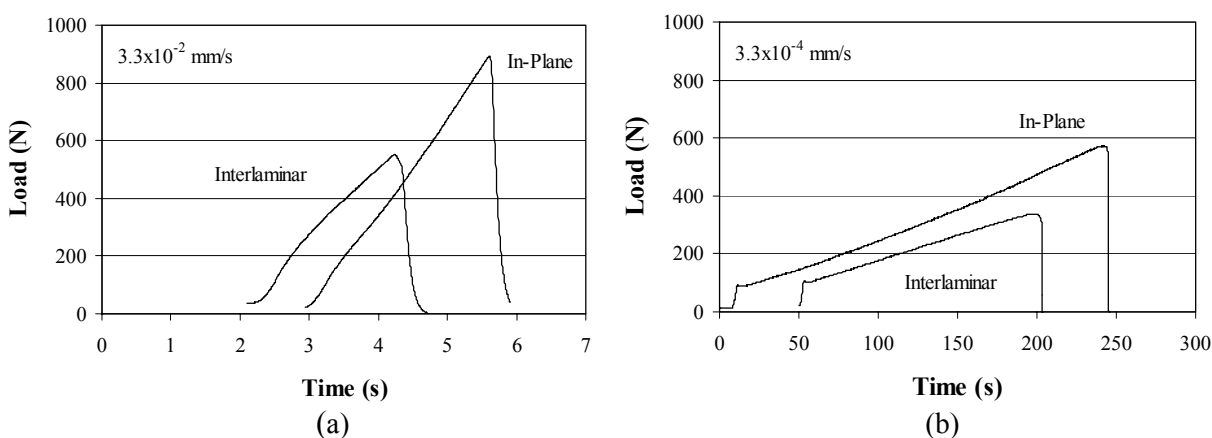


Figure 5. Typical examples of load versus time curves of SiC/BSAS composite tested in interlaminar and in-plane shear at 1100 °C in air at: (a) fast test rate of 3.3×10^{-2} mm/s, and (b) slow test rate of 3.3×10^{-4} mm/s.

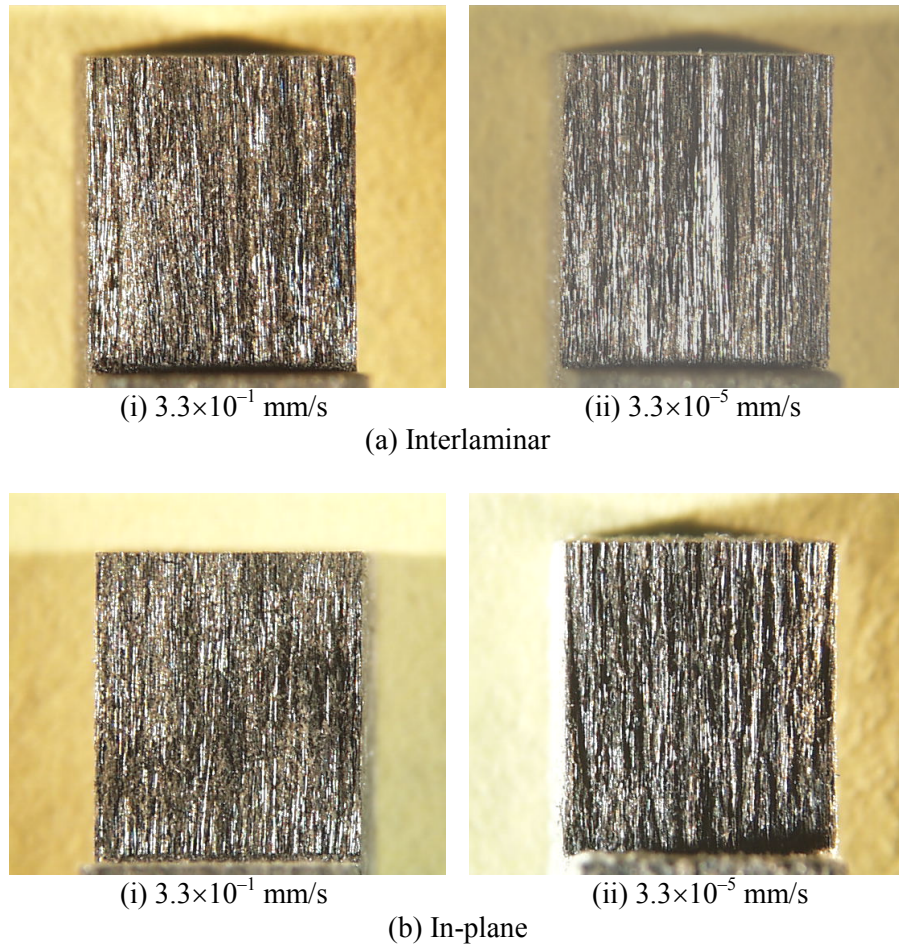


Figure 6. Typical examples of fracture surfaces of SiC/BSAS composite at high (3.3×10^{-1} mm/s) and low (3.3×10^{-5} mm/s) test rates, tested in shear at 1100 °C in air: (a) interlaminar and (b) in-plane shear specimens.

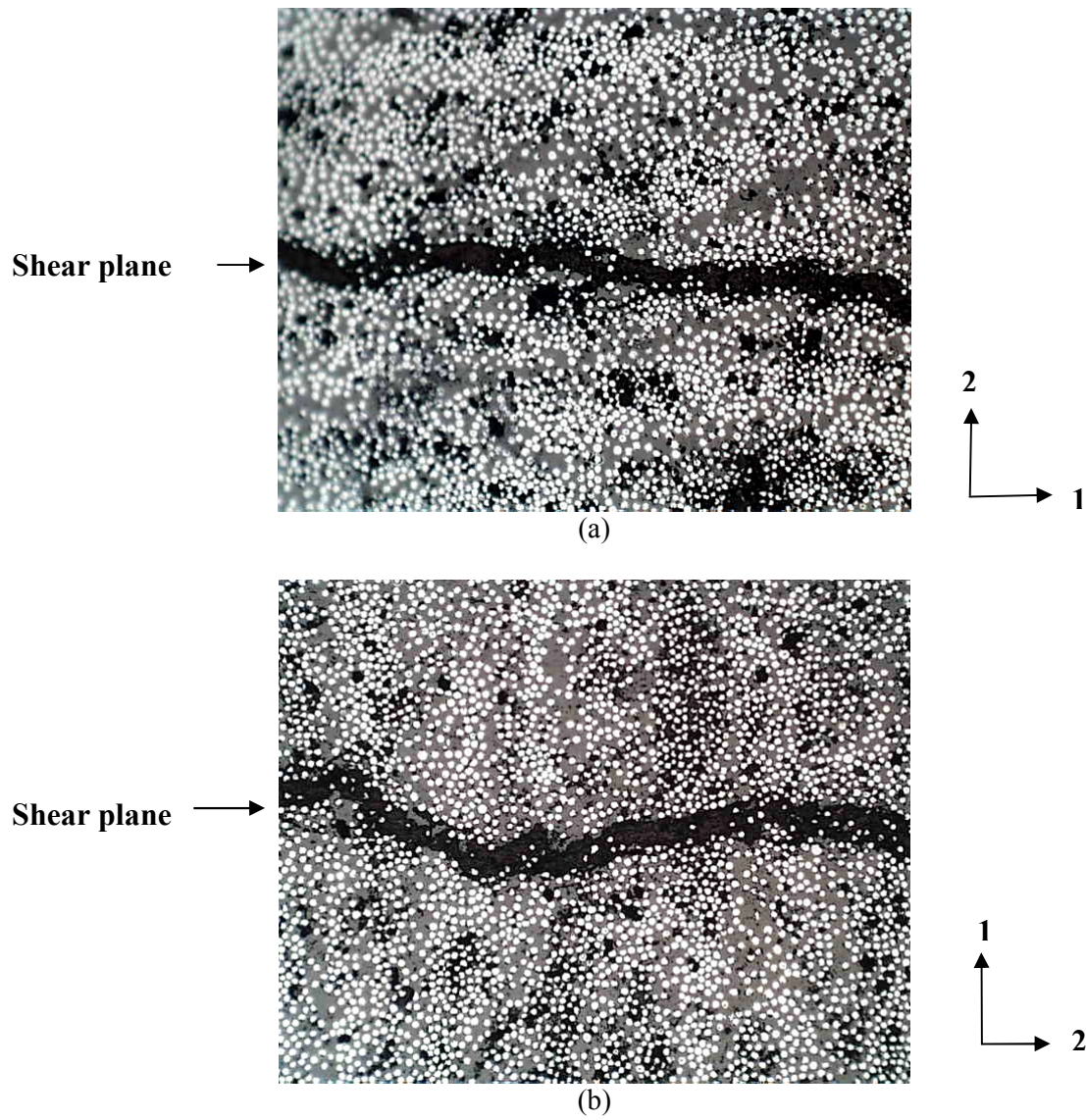


Figure 7. Cross sectional views showing shear planes, normal to fiber direction, of SiC/BSAS composite tested in shear at 1100 °C in air: (a) interlaminar and (b) in-plane specimens.

This also can be seen from planes perpendicular to fiber direction, in which shear planes were reasonable straight in interlaminar specimens but were more tortuous in in-plane specimens, as shown in Figure 7. A more detailed fractography is underway to better understand the mechanisms associated with shear failure in conjunction with SCG and material direction.

DISCUSSION

The strength degradation in shear with decreasing test rate exhibited by the SiC/BSAS composite in this work was very similar to that in tension shown by not only CFCCs such as SiC/CAS, SiC/MAS, SiC/SiC, C/SiC, SiC/BSAS (2-D) [6,7,9] but advanced monolithic ceramics such as silicon nitrides, silicon carbides and aluminas [10]. The strength degradation in tension with decreasing test rate for advanced monolithic ceramics is known as a slow-crack growth (“dynamic fatigue”) phenomenon, commonly expressed by the following empirical power-law relation [11]

$$v = \alpha (K_I / K_{Ic})^n \quad (2)$$

where v , K_I and K_{Ic} are crack velocity, stress intensity factor and fracture toughness under mode I loading, respectively. α and n are called slow crack growth (SCG) parameters in mode I. Based on this power-law relation, the tensile strength (σ_f) can be derived as a function of applied test rate or stress rate ($\dot{\sigma}$) with some mathematical manipulations [12–14].

$$\sigma_f = D [\dot{\sigma}]^{1/(n+1)} \quad (3)$$

where D is another SCG parameter associated with inert strength, n and crack geometry. Equation (3) can be expressed in a more convenient form by taking logarithms of both sides

$$\log \sigma_f = \frac{1}{n+1} \log \dot{\sigma} + \log D \quad (4)$$

A test methodology based on Equation (3) or (4) is called constant stress-rate (“dynamic fatigue”) testing and has been established as ASTM Test Methods (C1368 [13] and C1465 [14]) to determine SCG parameters of advanced monolithic ceramics at ambient and elevated temperatures. As mentioned before, the data fit to Equation (4) has shown very reasonable even for CFCCs including SiC/CAS (1-D), SiC/MAS (1-, 2-D), SiC/SiC (2-D), C/SiC (2-D), and SiC/BSAS (2-D) in tension at 1100 to 1200 °C in air. This indicates that SCG or damage evolution/accumulation or delayed failure of those composites would be adequately described by the power-law type relation, Equation (1). The SCG parameter n is the most important life prediction parameter since it controls a measure of susceptibility to slow crack growth or damage evolution/accumulation and in turn to strength degradation, as implied from Equation 2. For monolithic ceramics, glasses or glass-ceramics, the susceptibility to SCG is typically categorized such that significant SCG lies in the range of $n < 30$, intermediate SCG in $n = 30-40$, and insignificant SCG is in the range of $n \geq 50$. For the aforementioned composites, the values of n were all within $n = 10-20$, hence, the composites can be termed ‘highly’ susceptible to SCG or damage evolution/accumulation. Typical toughened modern monolithic silicon nitrides at 1200 °C exhibit $n \geq 50$. Therefore, the CFCCs exhibited a significantly greater susceptibility to SCG or damage evolution/accumulation than toughened monolithic silicon nitrides. Examples showing invariably significant strength degradation and consequently significant SCG susceptibility for some CFCCs in constant stress-rate testing in tension [6,7] are presented in Figure 8.

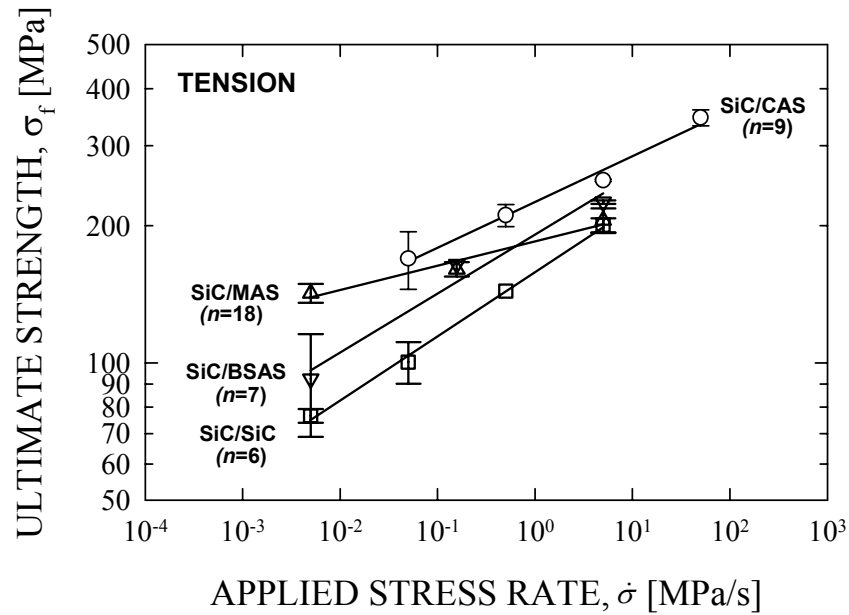


Figure 8. Examples of ultimate tensile strength as a function of test rate at elevated temperatures in air for some CFCCs including SiC/MAS (2-D; 1100 °C), SiC/CAS (1-D; 1100 °C), SiC/SiC (2-D woven; 1200 °C), and SiC/BSAS (2-D; 1100 °C) [6,7].

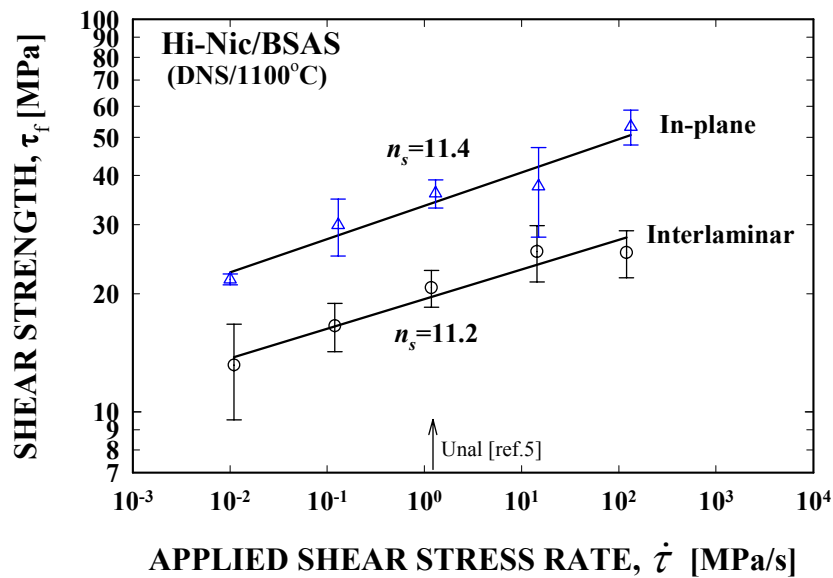


Figure 9. Shear strength as a function of applied shear stress rate for SiC/BSAS composite at 1100 °C in air, reconstructed from the data in Figure 4 based on Eq. (10). The arrow in the ‘stress-rate’ axis indicates the stress rate used by Ünal and Bansal [5].

Based on the experimental results of the SiC/BSAS composite showing the dependency of shear strength on test rate, the reasonable data fit to log-log relation, and on the indication of SCG from the load-vs-time curves, the governing failure mechanism in shear can be assumed to be the one associated with SCG, similar in expression to the power-law relation of Equation (2). Hence, the following empirical slow crack velocity formulation is proposed here in shear, modified from the one (Equation 2) in tension,

$$v = \frac{da}{dt} = \alpha_s (K_{II} / K_{IIc})^{n_s} \quad (5)$$

where v , a , t , K_{II} and K_{IIc} are crack velocity, crack size, time, mode II stress intensity factor, and fracture toughness in mode II loading, respectively. α_s and n_s are slow crack growth (SCG) parameters in shear. In monotonic shear testing, constant displacement rate or constant load rate can be employed to test specimens so that shear stress applied to test specimens is a linear function of test time

$$\tau = \int_0^t \dot{\tau}(t) dt = \dot{\tau} t \quad (6)$$

where τ is shear stress, and $\dot{\tau}$ is applied shear stress rate determined from $[\dot{\tau} = P/(WL_n t)]$ from load versus time curves in displacement control or directly from $[\dot{\tau} = \dot{P}/WL_n]$ in load control with $\dot{P}$ being load rate applied through a test frame. The generalized expression of stress intensity factor in shear for the case of an infinite body with a semicircular crack takes the form [15]

$$K_{II} = Y_s \tau a^{1/2} \quad (7)$$

where Y_s is a crack geometry factor, typically $Y_s = 1/(2\sqrt{\pi})$. Using Equations (5) to (7) and following a similar procedure as used to drive Equation (3) in mode I, one can obtain shear strength (τ_f) as a function of applied shear stress rate as follows:

$$\tau_f = D_s [\dot{\tau}]^{1/(n_s+1)} \quad (8)$$

where

$$D_s = [B_s(n_s + 1) \tau_i^{n_s-2}]^{1/(n_s+1)} \quad (9)$$

where $B_s = 2K_{IIc}/[\alpha_s Y_s^2 (n_s - 2)]$ and, τ_i is the inert strength that is determined in an appropriate inert environment or at a fast test rate whereby no or little SCG occurs. Equation (8) can be expressed in a more convenient form by taking logarithms of both sides

$$\log \tau_f = \frac{1}{n_s + 1} \log \dot{\tau} + \log D_s \quad (10)$$

which is identical in form to Equation (3) of mode I loading. SCG parameters n_s and D_s in shear can be determined from slope and intercept of a linear regression analysis of the log (*individual shear strength*) versus log (*individual shear stress rate*) data based on Equation (10). The SCG parameter α_s can be estimated from Equation (9) with appropriate constants and parameters associated.

Figure 9 shows the results of shear strength as a function of applied shear stress rate plotted based on Equation (10), with displacement rate being converted to shear stress rate. The slow crack growth parameters were determined as $n_s = 11.2$ and $D_s = 19.24$ for interlaminar direction and $n_s = 11.4$ and $D_s = 33.27$ for in-plane direction. The corresponding coefficients of correlation (r_{coef}) of curve fit were 0.8442 and

0.8850, respectively, for interlaminar and in-plane directions, showing reasonable data fit to the equation.* It is noteworthy that the value of n_s , a measure of susceptibility to SCG, was identical to each other, regardless of material direction. The value of SCG parameter n_s (=11) in shear also compares reasonably with that of n (=7) in tension for the similar 2-D SiC/BSAS composite tested at 1100 °C in air [7], indicating that the SiC/BSAS composite exhibited a significant susceptibility to SCG in shear as well. In general, the patterns of SCG for many monolithic advanced ceramics tested at elevated temperatures are well defined in fracture surfaces in terms of its configuration and size. By contrast, the patterns of SCG for CFCCs subjected to either in tension or in shear are not obvious and rather obscured by the nature of composite architecture so that it would be very difficult and/or a great challenge to derive a definite evidence of SCG from overall fracture surface examinations. Hence, the method proposed here –shear strength degradation with respect to test rate through constant stress-rate testing- would be a simple, quick and convenient way to check and quantify the degree of SCG in shear for CFCCs at elevated temperatures. The residual glassy phase may have been a major cause of SCG in the SiC/BSAS composite under shear.

Constant stress rate testing in tension has shown as a possible alternative to life prediction testing, as verified with stress rupture (constant stress) testing for various CFCCs at elevated temperatures at 1100 to 1200 °C [6,7]. This indicated that the same failure mechanism might have been operative, independent of loading configuration that was either in monotonic loading (constant stress rate) or in static loading (constant stress). Therefore, it is expected that a single failure mechanism, SCG, would be dominant in shear for the SiC/BSAS composite regardless of loading configuration and that life prediction in shear from one loading configuration to another could be made analytically or numerically depending on the complexity of loading configurations. A phenomenological, simplified life prediction is proposed based on the following relation that was modified to account for shear loading from the relation primarily used for brittle monolithic ceramics in tension [16]

$$t_f = \left[\frac{D_s^{n_s+1}}{n_s + 1} \right] \tau^{-n_s} \quad (11)$$

where t_f is the time to failure, τ is applied shear stress, and n_s and D_s are SCG parameters determined for each material direction in constant stress rate testing. The resulting life prediction curves in shear are shown in Figure 10 for both interlaminar and in-plane directions. Since the SiC/BSAS composite used in this work was no longer available, verification of the prediction by conducting constant stress (“stress rupture”) testing was not feasible so that only a method of prediction was proposed. An alternative composite is being sought for a future work of verification. It has been shown previously that under tension loading the overall life prediction of CFCCs was in reasonable agreement at least for a short period of lives with experimental data despite that the SCG formulation (Equation (1)) was phenomenological in nature without taking into account fiber/matrix architecture and interfacial microscopic mechanics of the composites.

The results of shear strength behavior of the 1-D SiC/BSAS showed that constant stress-rate testing, commonly utilized in determining life prediction parameters of monolithic ceramics in tension, could be applicable to determine phenomenological life prediction parameters of the composite material even in shear. This is also indicative that the overall failure mechanism in shear would be the one governed by the power-law type of slow crack growth. The merit of constant stress rate testing, incorporated with the power-law formulation, is enormous in terms of simplicity, test economy (short test times), and less

* If average shear strength and average shear stress rate were used, the coefficients of correlation were $r_{cof}=0.9708$ and 0.9723 , respectively, for interlaminar and in-plane directions. The corresponding SCG parameters were found to be $n_s=12$ and $D_s=19.4$ and $n_s=11$ and $D_s=33.5$, respectively, for interlaminar and in-plane directions. Hence, the difference in SCG parameters between individual data and average data approaches was negligible; however, the difference in coefficient of correlation was somewhat amplified. It is generally recommended that individual data approach be used in regression analysis for improved statistical accuracy [13,14].

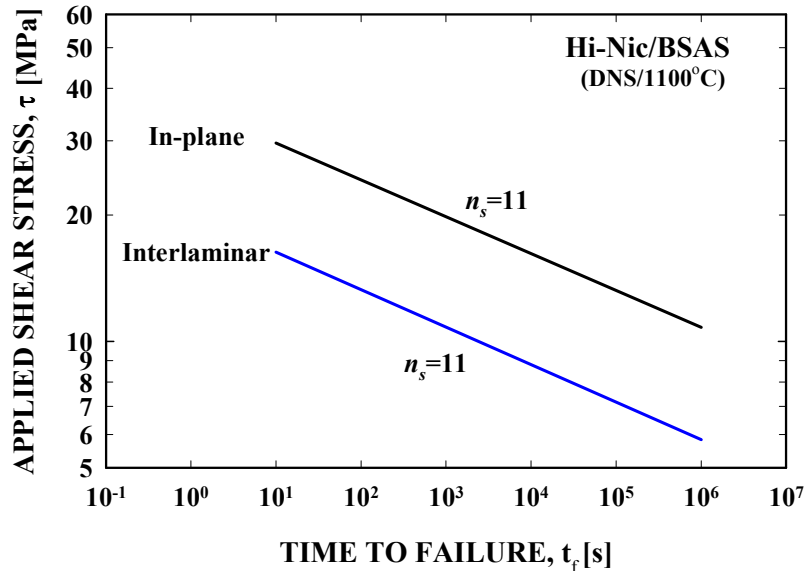


Figure 10. A life prediction diagram of SiC/BSAS composite at 1100 °C under constant stress condition, constructed from the data of Figure 9 based on Eq. (11). Note that the diagram represents at a failure probability of approximately 50 percent.

(strength) data scatter over other stress rupture or cyclic fatigue testing. Although the experimental results and phenomenological SCG model were presented in this work, a more detailed study regarding microscopic shear failure mechanisms associated with matrix/fiber interaction, matrix cracking and its effect on slow crack growth, and environmental effects, etc, [17–20] is still needed. Finally, the results of this work also suggest that care must be exercised when characterizing elevated-temperature shear strength of composite materials. This is due to that fact that elevated-temperature shear strength has a relative meaning if a material exhibits rate dependency: the shear strength simply depends on which test rate one chooses. Therefore, at least two test rates (high and low) are recommended to better characterize high-temperature shear strength behavior of a composite material, as suggested previously for the determination of ultimate tensile strength of CFCCs at elevated temperatures [6,7].

CONCLUSIONS

The interlaminar and in-plane shear strengths of a unidirectional Hi-Nicalon™ fiber-reinforced barium strontium aluminosilicate (SiC/BSAS) composite were determined at 1100 °C in air as a function of test rate. For a given test rate, the interlaminar shear strength was approximately 70 percent greater than the in-plane shear strength. The composite's shear strength exhibited a significant effect on test rate, regardless of orientation which was either in interlaminar or in in-plane direction, resulting in significant shear-strength degradation of about 50 to 60 percent as test rate decreased from 3.3×10^{-1} mm/s to 3.3×10^{-5} mm/s. The rate dependency of shear strength was analogous to that of ultimate tensile strength at 1100 °C observed in a similar composite material (2-D SiC/BSAS) in which tensile strength decreased by about 60 percent when test rate varied from the highest to the lowest. A phenomenological power-law slow crack growth formulation was proposed to take account for shear-strength degradation behavior of the composite. Furthermore, constant shear-stress rate testing was proposed as a possible means of life

prediction testing methodology to determine life prediction parameters of the composite in shear at elevated temperatures.

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REPORT DOCUMENTATION PAGE			Form Approved OMB No. 0704-0188	
Public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden, to Washington Headquarters Services, Directorate for Information Operations and Reports, 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302, and to the Office of Management and Budget, Paperwork Reduction Project (0704-0188), Washington, DC 20503.				
1. AGENCY USE ONLY (Leave blank)		2. REPORT DATE April 2003		3. REPORT TYPE AND DATES COVERED Technical Memorandum
4. TITLE AND SUBTITLE Dependency of Shear Strength on Test Rate in SiC/BSAS Ceramic Matrix Composite at Elevated Temperature			5. FUNDING NUMBERS WBS-22-714-04-30	
6. AUTHOR(S) Sung R. Choi, Narottam P. Bansal, and John P. Gyekenyesi				
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) National Aeronautics and Space Administration John H. Glenn Research Center at Lewis Field Cleveland, Ohio 44135-3191			8. PERFORMING ORGANIZATION REPORT NUMBER E-13783	
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) National Aeronautics and Space Administration Washington, DC 20546-0001			10. SPONSORING/MONITORING AGENCY REPORT NUMBER NASA TM-2003-212182	
11. SUPPLEMENTARY NOTES Sung R. Choi, Ohio Aerospace Institute, Brook Park, Ohio 44142; Narottam P. Bansal and John P. Gyekenyesi, NASA Glenn Research Center. Responsible person, Sung R. Choi, organization code 5920, 216-433-8366.				
12a. DISTRIBUTION/AVAILABILITY STATEMENT Unclassified - Unlimited Subject Category: 07 Available electronically at http://gltrs.grc.nasa.gov This publication is available from the NASA Center for AeroSpace Information, 301-621-0390.			12b. DISTRIBUTION CODE	
13. ABSTRACT (Maximum 200 words) Both interlaminar and in-plane shear strengths of a unidirectional Hi-Nicalon™ fiber-reinforced barium strontium aluminosilicate (SiC/BSAS) composite were determined at 1100 °C in air as a function of test rate using double notch shear test specimens. The composite exhibited a significant effect of test rate on shear strength, regardless of orientation which was either in interlaminar or in in-plane direction, resulting in an appreciable shear-strength degradation of about 50 percent as test rate decreased from 3.3×10^{-1} mm/s to 3.3×10^{-5} mm/s. The rate dependency of composite's shear strength was very similar to that of ultimate tensile strength at 1100 °C observed in a similar composite (2-D SiC/BSAS) in which tensile strength decreased by about 60 percent when test rate varied from the highest (5 MPa/s) to the lowest (0.005 MPa/s). A phenomenological, power-law slow crack growth formulation was proposed and formulated to account for the rate dependency of shear strength of the composite.				
14. SUBJECT TERMS Ceramic composites; SiC/BSAS; Elevated-temperature shear strength; Slow crack growth; Mechanical testing; Lifting			15. NUMBER OF PAGES 21	
			16. PRICE CODE	
17. SECURITY CLASSIFICATION OF REPORT Unclassified	18. SECURITY CLASSIFICATION OF THIS PAGE Unclassified	19. SECURITY CLASSIFICATION OF ABSTRACT Unclassified	20. LIMITATION OF ABSTRACT	